

## **PRODUCTION OF MAGNESIUM IN 30KA CELLS**

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A prototype plant for the production of magnesium metal by fused salt electrolysis of anhydrous  $\text{MgCl}_2$  has been set up during 1985 at DMRL, Hyderabad, as a joint development project with CECRI, Karaikudi. Cells of 30 KA capacity have been designed, fabricated and erected. A suitable absorption system has been installed for the treatment of generated chlorine.

The plant has been commissioned and one cell has been operated over a period of four months. The metal at the rate of 100 - 150 kg/day with a current efficiency of over 70% has been obtained. The metal on remelting has been found to be of 99.9% purity.

The paper describes the plant and the process units. The experimental results and the performance of the cell are discussed.

**Key words:** Magnesium electrowinning, magnesium chloride electrolysis

### **DESCRIPTION OF FACILITY**

The magnesium pilot plant consists of cell house, power supply system, chlorine disposal system and power control units. Two 30 KA cells and the process control panel are located in the cell house. The power supply system consisting of AC transformer, DC source (30 KA, 0 to 15 V) and a circuit breaker are housed in the rectifier room. Chlorine disposal system comprises of chlorine pipelines, blowers to evacuate chlorine from the cells, lime slurry tanks, FRP towers and pumps for circulation of slurry. The cell design is based on the modular concept developed at Central Electrochemical Research Institute (CECRI) who have earlier operated a 8 KA cell with 4 modules [1].

Each cell assembly consists of twelve 2.5 KA modules arranged in three rows of four each. 150mm dia. graphite anodes are suspended between the cathodes from the cell top. The cathode is mild steel frame forming an octagon around the anodes. The effective height of this grid assembly is 900 mm. The graphite anodes are fixed on to aluminium bus bars, enter through the lid and have common electrical terminals. Similarly the cathodes have also common electrical terminals. The distance between the anode and cathode is 22-27mm.

The cell body is lined with high density alumina bricks. A refractory curtain is provided above the cathode level and between electrolytic and nonelectrolytic zones of the cell. The curtain avoids the entry of chlorine to the metal ladling zones. Provisions have also been made for easy removal of sludge from the base, addition of salt mixture, collection of metal and suction of chlorine. The hoppers are provided at the cell lid to facilitate addition of magnesium chloride to the bath.

The chlorine from the cell is sucked off by a blower and a ventury placed between the cell and the FRP towers connected by 150mm duct with junction boxes. Calcium hydroxide slurry is prepared in concrete tanks and circulated alternatively in one of the two

towers by slurry pumps. The absorption unit is designed for 2 tonnes of chlorine per day.

### **EXPERIMENTAL**

#### **Start up of the cell**

Initial melting of the salt-mixture consisting of  $\text{NaCl} + \text{KCl} + \text{BaCl}_2$  is carried out by A.C. heating. The two auxiliary A.C. electrodes provided in the cell are shorted by a graphite wedge. By gradual addition of the above salt mixture on to the red hot-wedge, a molten pool of electrolyte is formed and built up to the desired level. At this stage required amount of anhydrous magnesium chloride in small lumps is fed into the cell, to get the operating level. D.C. power is impressed then.

#### **Feeding cycle and level control**

Electrolysis is continued by periodic addition of  $\text{MgCl}_2$ , maintaining the desired bath composition and the level. The quantity of the chloride feed is decided on the basis of the metal output and the bath composition is maintained by periodical analysis.

The cell being modular, it derives the advantage of the hydrodynamic effect of chlorine evolution in the electrolytic zone which enables the magnesium metal formed at the cathodes to be pushed away into the relatively tranquil nonelectrolytic zone. This passage of magnesium would be rendered difficult if the level of the electrolyte falls down below the cathode top. Under such situations the magnesium formed will continue to float in the electrolytic zone between the cathodes and anodes and will be subjected to recombination with chlorine. It is therefore essential to ensure that the level of the electrolyte under no circumstances falls below the cathode top. Too high a level, on the other hand, is also not desirable since it results in cooling of the electrolyte. A level of 2 to 8 cm above cathode top suits the operation ideally. Feeding cycles are fixed keeping the above points in mind in

addition to the fact that magnesium chloride concentration has normally to be maintained between 10 and 15%

These globules of metallic magnesium are pushed towards the nonelectrolytic zone and coalesce into a pool of metal. The metal is periodically ladled out and cast in mild steel moulds. The metal normally is covered by a thin layer of electrolyte preventing its contact with atmospheric air and thus does not burn. The chance of metal burning is however totally eliminated by dusting sulphur powder while pouring the metal. After cooling the metal, the ingot is removed from the mould.

The cell is provided with a slope towards the front (ladling side) so that the sludge is accumulated at the bottom of the nonelectrolytic zone. The sludge is removed using perforated ladles at least once in twenty four hours. Accumulated sludge acts as resistor and part of the current impressed is utilised for its heating, resulting in decreased current efficiency and overheating of the cell.

#### Separation and absorption of chlorine

Chlorine gas coming from the cell at 400°C is sucked through a venturi where it gets mixed with air attaining a temperature of around 60°C. It goes to the surge drum where chlorine and air mixing is completed and desired pressure is maintained. A blower provides the required pressure (20" water column), to introduce the chlorine - air mixture at the bottom of the absorption tower. Milk of lime is sprayed from the top of the absorption tower which falls through baffle plates. The air, free of chlorine, goes out from the top of the tower. When the lime slurry in the circulation tank gets saturated with chlorine, the ascend tower is put into operation.

#### MODIFICATIONS

##### Anode assembly

Graphite anodes originally used were of 150mm dia and the interelectrode distance was 22mm. Each anode was made by joining two 1200mm long graphite rods by means of nipples. This resulted in slight deviation in the verticality of the anode thereby resulting in shorting with cathode. Therefore the diameter of the lower portion of the anode was reduced to 140mm.

During the initial trial it was found that the graphite rods just below the lid were eroded probably due to the air leaks through the gaps between graphite anodes and the circular openings of the lid through which the rods pass. This was controlled to a large extent by proper packing and filling up with refractory cement around the anodes above the lid after making a suitable tray arrangement. The portions of the graphite rods between the bath level and the lid were also coated with refractory cement. This resulted in prolonging the life of anodes.

##### Chlorine line

The pipelines consisting of 150mm dia stoneware pipes from cells to the venturi have been coated with acid proof cement from inside and refractory cement from outside. Manometers are incorporated at all the junctions to see the suction head. There have been leakages in the line at the stoneware pipe joints from venturi to the absorption unit. This was replaced by a single piece FRP line. The number of bends in the line were also minimised to improve suction.

#### RESULTS

The cell has been operated over a period of four months including a continuous run of two months. Typical values obtained for cell voltage, current density, current efficiency and metal output are given in Table I.

Table I : Typical cell characteristics

Current (KA)	20-22
Rectifier voltage (V)	5.4-5.7
Cell voltage (V)	4.5-4.7
Interpolar gap (mm)	22 - 27
Current density (A/cm <sup>2</sup> )	0.6
Current efficiency (%)	65-75
DC consumption (kWh/kg)	15-17
Metal output kg/day	125

From this it is observed that the difference between the rectifier voltage and cell voltage i.e. drop in the busbars is approximately one volt. The calculated voltage drop for the busbars alone at room temperature comes to around 0.5 V. However, taking into consideration the joints and junctions, and the rise in temperature of the busbars during the operation, a total drop of about one volt is reasonable.

The rectifier unit has been designed to provide 30KA at 0-15V. In operation, however, it could only deliver 22KA at 6V. Hence electrolysis experiments have been carried out at 4.5 - 6.0 volts, 22 KA current. A total of 11.5 tonnes of magnesium metal has been produced. Table II gives the analytical results of the remelted metal which conform to the standard specifications.

Table II: Analytical report of magnesium metal

	ASTM 9980A	DOW Mg4	DMRL
Aluminium	—	0.003% Max.	0.003% Max.
Copper	0.02% Max.	0.004% Max.	0.001%
Iron	—	0.040% Max.	0.030%
Manganese	0.01% Max.	0.006% Max.	0.005%
Nickel	0.001% Max.	0.001% Max.	0.001%
Silicon	—	0.005% Max.	0.005% Max.
Titanium	—	0.002% Max.	0.001% Max.
Zinc	—	0.003% Max.	0.001%
Lead	0.01% Max.	0.005% Max.	0.005% Max.
Magnesium	99.80% Min.	99.90 Min.	99.90 Min.

#### CONCLUSIONS

Based on the experimental experience and the data obtained, it can be stated that:

- (i) The design of the electrolytic cells, busbars, chlorine absorption unit and the operating parameters are suitable and optimum for continuous production of the metal at a rate of around 200 kgs per day with 30 KA DC power supply.

(ii) The quality of the metal produced conforms to the specifications for electrolytic magnesium.

The facility set up and the expertise developed are expected to be very useful for the future magnesium plants in the country.

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